United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program

SOP No.: PDP-QC-12		Page 1 of 5
TITLE: Unidentified Analytical Responses		
Revision: Original	Replaces: NA	Effective: 2/1/96

1. Purpose:

To provide a standard method for processing unidentified analytical responses (UARs), which may occur during pesticide analysis of commodities in USDA-AMS-Pesticide Data Program (PDP) laboratories.

2. Scope:

This SOP shall be followed by all analytical laboratories conducting residue studies for PDP. This includes laboratories conducting stability and other studies which may impact PDP.

3. Outlines of Procedure:

5.1 Definition of UAR
5.2 Recognizing UARs
5.3 Data Acquisition of UARs in PDP laboratories
5.4 PDP Policy for Processing UARs

4. References:

Laboratory Communication No. Science Division (SD) 02; Unidentified Analytical Responses (UARs) - Pesticides Amendment No. 3
Laboratory Communication No. SD 02; Unidentified Analytical Responses (UARs) Pesticides Amendment No. 2
Personal Communication, Bob Epstein, USDA-AMS-PDP

5. Specific Procedures:

This Standard Operating Procedure (SOP) describes PDP policy for processing UARs generated during organochlorine (OC), organophosphate (OP), organonitrogen (ON),

organosulfurs (OS) and carbamate (CAR) tests performed on PDP commodities using methods approved by the Residue Branch in Manassas, VA.

All UARs found during the analysis of chlorophenoxy acid herbicides, avermectin, and formetanate hydrochloride by PDP laboratories shall be reported to the Residue Branch in Manassas, VA. Compounds such as dicamba, dalapan, MCPA, and 2,4,5-T can be easily recognized in the acid herbicide screen. The Residue Branch will maintain all UAR records and notify the states, if additional action is required.

5.1 Definition of UAR

Unidentified Analytical Response (UAR) is a chromatographic response observed in a specific detection system. This response is caused by a compound with chromatographic properties different from those of known reference pesticide standards and reagents, as well as integral components of commodity matrices.

5.2 Recognizing UARs

A UAR in fruits and vegetables is any response (as defined in section 5.1), which appears anywhere in a chromatogram (from the solvent front to the end of chromatogram) that produces an area or peak height greater than the response produced by the associated process control standard (s) at the 5 X LOQ concentration.

The total elapsed time for a chromatogram produced from injection of fruits and vegetables extracts should be sufficiently long to preclude a UAR from appearing in a subsequent chromatogram produced by injection of another sample extract.

All chromatographic data such as labeled chromatograms of samples, standards, blanks and recovery data used for proper quantitation of UAR will be filed with appropriate UAR identification (such as UARPDP-1), and stored at the laboratory in a manner that it can be retrieved, when necessary.

5.3 Data Acquisition of UARs in PDP Laboratories

RRT (relative retention time) corresponds to the total time a UAR takes relative to the time taken by the process control standard, when a sample extract with a UAR is injected into a chromatograph and allowed to pass through a column. Determine RRT by dividing the UAR retention time by the retention time for the process control standard, calculated to two decimal places.

RCR (relative concentration response) corresponds to the concentration of the UAR relative to the concentration of the process control. Determine the UAR's RCR by multiplying the peak height (or area) of the UAR by the concentration of the process control standard used, divided by the peak height or (area) for the process control standard. If the analysis is repeated, the mean of two values from chromatographic data is used to report RCR to two decimal places.

5.4 PDP Policy for Processing UARs

The State laboratories shall record information on all UARs in a recording log designated for this purpose. This log shall maintain such information as sample identification number, frequency of its occurrence, Relative Retention Time (RRT), Relative Concentration Response (RCR), worksheet for UAR identification, or any other information (MSD, Ion Trap, AED), which may help determine its identity and the reason for this response.

Once the analysis and the data acquisition are completed, and the UAR cannot be identified, the States shall inform the Residue Branch in Manassas, VA. The Residue Branch will check its existing records on UARs to determine if UAR in question has been reported by another State or in another commodity.

If the compound(s) is recognized through a computer search of the system library or other mass spectrometry sources, the compound(s) shall be reported to the Residue Branch with appropriate means of identification and other required data. If the UAR identified is a pesticide not currently in PDP, it may be recommended for inclusion in the PDP monitoring after a decision is made by the USDA-AMS-PDP in consultation with EPA.

If the UAR remains unidentified, the Residue Branch will make arrangements for

the sample to be shipped to California Department of Food and Agriculture (CDFA) for confirmation of UAR with triple stage mass spectrometry. The Residue Branch will provide CDFA with the available analytical information, along with the computer search data from the system library or other mass spectrometry literature sources.

If additional information, such as full scan electron impact mass spectrometer, positive ion chemical ionization mass spectrometry using methane, and elemental characterization by GC/AED,is requested by CDFA, the Residue Branch will make efforts to provide the requested information available to CDFA.

CDFA will perform GC-Negative Ion Chemical Ionization mass spectrometry (GC/NICI/MS), GC/MS/MS and /or LC/MS/MS means of identification, if the compound is suitable for both technologies. CDFA will provide the Residue Branch results of their GC/MS/MS and /or LC/MS/MS findings.

If the UAR is identified as a pesticide not currently in PDP, it may be recommended for inclusion in the PDP monitoring after a decision is made by the Residue Branch. The Residue Branch will retain information on UARs submitted by CDFA for future reference.

Martha Lamont *2/1/96*

Approved By: Martha Lamont Date
Technical Director, PDP

USDA/AMS
8700 Centreville Road, St. 200
Manassas, VA 22110
(703) 330-2300

Robert Epstein *2/1/96*

Approved By: Robert Epstein Date
Program Administrative Director, PDP

USDA-AMS SD
P.O. Box 96456-3525
South Building
Washington, DC 20090-6456
(202) 720-2158